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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
841 Chestnut Building
Philadelphia, Pennsylvania 19107

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SUBJECT: Standard Chlorine of Delaware, Inc. DATE: 9/17/92

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TO: File through
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On August 27, 1992 I conducted a RCRA Compliance Inspection at Standard Chlorine of Delaware, Inc. (SCD) in Delaware City, Delaware. With me on this Inspection was Alan Simpson, Environmental Scientist II, for the Delaware Department of Natural Resources and Environmental Control (DNREC). The Facility representatives for this inspection were Paul Johnston, Environmental Manager, and Robert Touhey, Vice President Environmental Affairs for SCD. All process descriptions and other information about the plant were obtained through discussions with the Facility representatives and through direct observation.

SCD manufactures various chlorinated benzenes for industrial uses, such as electrical insulating fluids to replace PCB's in reconditioned transformers and as intermediates to the production of pesticides and pharmaceuticals. The raw materials used in the process are benzene, received in tank truck quantities, chlorine, received either in tank cars or via a pipeline from Oxychem nearby, and iron, in the form of ferric chloride. The material is produced via a Friedel-Crafts type reaction conducted as a continuous batch process. The Facility is divided into the "main plant" which has four (4) distillation/fractionation columns for the production of mono- and dichlorobenzene, and a smaller "plant" for the production of 1,2,4-trichlorobenzene and a "polychlore" plant for the production of tetrachlorobenzenes.

Though all plants are similarly run, the Main plant is larger and more complex than the others because it uses the least refined raw materials. Benzene and chlorine are mixed in a reaction vessel in the presence of the ferric chloride catalyst and heated to approximately 40°C. The different types of chlorobenzenes are produced by directing the chemical reaction by changing the benzene/chlorine ratio, or adjusting the specific time and temperature of the reaction. It is not currently possible to limit this reaction to the production of monochlorobenzene. This reaction generally results in the

primary formation of monochlorobenzene, somewhat smaller amounts of mixed dichlorobenzenes and some residual trichlorobenzene as well as unreacted benzene. After reaction the reactor mass is stripped of unreacted benzene and directed to a static mixer where the reactor mass is washed with water (and sometimes caustic) to neutralize residual hydrogen chloride in the mixture (to be discussed below). The washed mixture is then passed through a dehydrator to remove residual water and then processed through a column to remove the monochlorobenzene. The remaining reactor mass is processed through two (2) columns to separate the dichlorobenzenes (predominantly the ortho- and para- isomers), and finally through a final column to separate out the trichlorobenzene. The para-dichlorobenzene is further refined in a crystallizer. The remaining materials are collected and are referred to by plant personnel as C2 bottoms.

The nature of the operations at SCD is such that the reaction chemistry and subsequent processing are not able to be precise. This means that reactions produce a mix of materials, not just the desired product, and that processing, such as distillation, does not provide for 100% recovery of the desired material. The C2 bottoms, for example, are a mixture of unrecovered trichlorobenzene, tetrachlorobenzene, and contaminants. These bottoms are accumulated in tank T34 (10,000 gallon capacity). As material is accumulated, it is assayed for trichlorobenzene content and, if sufficient amounts of TCE are present in the bottoms, the mass is recirculated back to the distillation column for further recovery of the trichlorobenzene. When the accumulated C2 bottoms exhibit insufficient trichlorobenzene levels for recovery the material is disposed of off-site as a hazardous waste, EPA waste code K085. The C2 bottoms are also tested for PCB's as it is not uncommon for up to 5000 ppm of PCB to show up in these bottoms.

Water removed from the dehydrator and the water from the static mixer washing step are combined in Tank 700 which serves as a surge capacity/feed tank to the T300/T301 distillation system. The combined waters are fed to tank T300 which feeds the column. The water is steam stripped of the organics, with the stripped water going to the Facility's on-site waste water treatment plant (WWTP) and the organics portion being returned to tank 700. When a sufficient organic layer develops in tank 700 it is decanted and returned to the process as part of the raw material feed.

Hydrogen chloride gas is generated as a by-product of this reaction and is removed from the reactor and passed to a scrubber to remove organic vapors (i.e., benzene) and then passed to a water unit to absorb the HCl and form a commercial grade

hydrochloric acid of 20 or 22 °Baume[1] (30% or 35% HCl). The acid is stored in twelve 30,000 gallon tanks in a tank farm designated as Area II.

The trichlorobenzene process is similar, but somewhat less involved due to the refined nature of the raw materials that are used in the process. Dichlorobenzene produced from the main plant is placed in the reactor with additional chlorine and a catalyst. A similar time/temperature controlled reaction takes place producing a mixture that is predominantly 1,2,4-trichlorobenzene with other trichlorobenzene isomers, tetrachlorobenzene isomers, and some higher polychlorinated benzenes comprising the remaining portion with the unreacted dichlorobenzene. The reactor mass is stripped of the unreacted dichlorobenzene and then washed with water (the water going to tank 700). The remaining charge is processed through a distillation column to separate the 1,2,4 trichlorobenzene isomer from the mixture. The resulting bottoms, consisting of approximately 95% tetrachlorobenzenes, 3% trichlorobenzenes and 2% others, is accumulated in tank T13A. T13A is assayed for trichlorobenzene content, and if sufficient, is recirculated back to the distillation column for recovery of additional trichlorobenzene. When the trichlorobenzene level is too low for recovery the column bottoms (designated as C3) are transferred to tank T13. SCD maintains an inventory of tetrachlorobenzenes[2] on-site as a product. This product material is stored in tanks T13 and T16 (30,000 gallon capacity). The C3 bottoms produced in excess of this product need is drawn off from tank T13A and placed into drums to be disposed of off-site as a hazardous waste, EPA waste code K085.

The Facility also has a production line for the synthesis of tetrachlorobenzenes which operates similarly to the other two processes. This process was not in operation at the time of this inspection.

In addition to the recycling of the C2 and C3 bottoms the Facility also operates a "scrap tank"; material contaminated with chlorinated benzenes, such as fullers earth, piping, clothing, and dirt is placed in this tank, which is filled with water and heated. The heated water releases the solidified materials from the scrap and suspends it so that it can be recovered. Fullers earth is a highly absorbent filter media that SCD uses to remove color from its finished materials as they are

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[1] A measurement on the Baume Scale, a hydrometer scale that separately covers liquids with specific gravities greater and less than 1. (per Webster's dictionary)

[2] Mixed tetrachlorobenzenes and C3 bottoms are the same material. The Facility has production capability for tetrachlorobenzenes, but maintains a limited inventory of the by-product tetrachlorobenzenes as a supply for its customers.

transferred to the containers (e.g., tanktruck) that will be used to ship them off-site. When spent, (i.e., clogged with the solidified chlorobenzenes) the filter "pots" are cleaned out and the material placed in drums to await processing in the scrap tank. After processing the fullers earth is drummed and disposed of off-site as D021/D027 hazardous waste.

SCD has one non-manufacturing operation at this site; the electro-chemical area. In this area the facility blends an electrical insulating fluid, using a mixture of tri- and tetra-chlorobenzenes, to replace the PCB oil commonly found in older transformers. SCD also sells a similar mixture to use for flushing PCB transformers that are to be reconditioned. As the Facility is only blending the fluid the only waste from this process area is filter bags from the tri- and tetra-chlorobenzenes.

The Facility has one main process stack, operated under permit #APC87035. All process vents and emissions are first passed through a caustic scrubber and then vented through this stack. The primary emission from this facility is nitrogen, which comes from blanketing the benzene tanks. The caustic used in the main scrubber is constantly replenished via a flow through system. The caustic is used in the main scrubber and then is passed onto other areas of the plant for use in smaller scrubbers and other areas. The spent caustic is eventually directed to the WWTP.

The Facility has a NPDES permit to discharge to the Delaware River (number not identified, but verified by the State) operating one process water outfall and two stormwater outfalls. The waste water treatment sludge is currently not handled as a hazardous waste as test results obtained by the facility show no excedences of hazardous waste characteristic levels.

During the Facility tour we found most areas of the plant to be well managed. There was not much to see as the processes are all hard piped through various tanks, reactors and columns, but the different process areas and associated reactors and columns were identified. An inspection of the "drum pad" found approximately 20 drums of material identified as containing contaminated glycol being stored prior to being reclaimed. Facility representatives stated that material was product contaminated with a coolant used for condensing purposes and was being reworked into the process as fast as practicable. To the north of the drum pad was a storage area where approximately 25 drums of the spent fullers earth were being stored. These drums were labeled with green non-hazardous waste labels, with an accumulation start date on them. However, these drums were not labeled as containing hazardous waste. None of these drums appeared have been in storage greater than 90 days. The Facility representatives stated that they did not feel that these materials were solid/hazardous wastes as they were being reclaimed, and that only the residuals from the reclamation was

actually a hazardous waste. However, as the fullers earth is a spent filter media and contains significant quantities of chlorobenzenes (i.e., greater than the TC limits) this material is properly classified as a hazardous waste, EPA waste code D021 and/or D027 depending on chlorobenzene content.

After observing the drum storage areas we walked the rest of the Facility observing various tankfarms and portions of the waste water treatment plant. Behind the northernmost tankfarm we observed the scrap tank. This unit is a large steel box with a close fitting screened box that fits inside. Scrap pieces are placed in the box and lowered into the water. After processing the box is raised to remove the scrap, which is drummed or placed in roll-off boxes for disposal off-site as a hazardous waste, D021 and/or D027.

Also observed were the bulk storage tanks and tank cars for the "crude tetrachlorobenzene mixture". According to the file record, in the late 1970's and early 1980's SCD had a market for the mixed tetrachlorobenzene left over from the trichlorobenzene process (the C3 bottoms, K085). In the early to mid 1980's SCD lost most of its market for the crude tetrachlorobenzene mix and was left with a large quantity of this material in storage. On site at this time, and since approximately 1982, is approximately 336,000 gallons of the mix, stored in tanks 315A, 326 and 327 (100,000 gallon capacity); two 18,000 gallon railcars; and three 10,000 gallon railcars. While this material may have been a product at one time (i.e., 1980) once SCD lost its market for this material it should have been managed as a waste. The material in storage is identical[3] to the material currently produced as C3 bottoms and disposed of as hazardous waste, K085.

An area of concern developed during the facility tour is the WWTP sludge. The reactor product washing step produces a wastewater, the listed waste K105, which is sent to the T700 tank for reclamation. Due to the closed loop design of the recycling/recirculation process this material is exempted from the definition of solid waste via 40 C.F.R. § 261.4(a)(8) and the analogous regulation in the current (1992) version of the Delaware regulations. The reclamation process produces a waste water which, according to Facility testing, does not exhibit a characteristic of hazardous waste. This wastewater is processed

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[3] I have to use the term "identical" a little loosely here. According to SCD some of the crude TCB mix was shipped to a Standard Chlorine facility in Kearny, NJ where additional distillation was conducted to remove di- and tri- chlorobenzene that was unable to be removed at the Delaware facility due to technical difficulties. After distillation, the material was returned to SCD. From the available description this material would be very similar to the C3 bottoms currently produced. This material was commingled with some of the materials in storage at SCD.

through the WWTP where it is further treated and any treatment residues become a component of the sludge from the WWTP. This sludge, according to Facility testing, does not exhibit any characteristics of hazardous waste. I have reviewed the listing document for the K105 waste, the preambles to the Federal Registers dealing with this closed-loop exemption, the pertinent guidance in the Permit Compendium and spoken with Ross Elliot of the Classification Section at EPA HQ and have determined that neither the wastewater from the reclamation process nor the WWTP sludge is a K105 listed hazardous waste. This is based on the belief that a listed hazardous waste can not be derived from the treatment of a nonhazardous waste. The scope of the listing for K105 (separated aqueous stream from the reactor product washing step in the production of chlorobenzenes), as presented in the listing background document, did not encompass reclamation residues that have been stripped of the chlorobenzene material. This is distinctly different from a waste such as F006 which seems to be the result of treating a nonhazardous waste[4] because this method of reclamation is designed to remove the constituents which were the basis for the listing of K105 waste; principally benzene, monochlorobenzene and dichlorobenzenes.

This situation is listed as an area of concern because the authorized version of the Delaware hazardous waste management regulations, dated June 22, 1984, do not contain an exemption analogous to that found in 40 C.F.R. § 261.4(a)(8). If this exemption is not considered the wastewater from the reclamation process and any resulting WWTP sludge would retain the K105 waste designation as these materials would be derived from the treatment of a listed hazardous waste. An equitable argument appears to exist as to whether SCD can be found to be in violation of the 1984 version of the Delaware regulations, the version EPA has authority to enforce in Delaware, when the conduct is in accordance with the current applicable State law and the existing Federal regulations. Therefore no violation was determined at this time.

The documentation review was limited to a review of the past years manifests. Wastes shipped off-site were C2 and C3 bottoms, K085; Tank clean out scale, D027; catch basin sludge, D018, D021, D027 and D036; and contaminated absorbent from transfer areas,

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[4] The listing for F006 waste, wastewater treatment sludges from electroplating operations, provides for a situation where the influent wastewaters may not exhibit any hazardous characteristic or a significant level of contamination for the constituents which were the basis for listing; in this case cadmium, hexavalent chromium, nickel and cyanides. In this instance EPA listed the residues derived from treating this potentially nonhazardous waste because the methods used in wastewater treatment tend to concentrate contaminates into the sludge, resulting in a hazardous waste being derived from the treatment of a non-hazardous waste.

various waste codes. The designated disposal facilities were Marine Shale Processors, Inc., Morgan City, Louisiana, benzene/chlorobenzene wastes; Rollins, Deerpark Texas, K085; ENSCO, El Dorado, Arkansas, K085; Safety-Kleen, New Castle Kentucky, K085; Safety-Kleen Corp., parts washers; and Pollution Control Industries of Indiana, East Chicago, Indiana, various wastes. No manifest related violations were documented. As SCD had been inspected by DNREC in December 1991 and Alan Simpson had reviewed the remaining RCRA required documentation at that time, other documentation, such as contingency plan and personnel training documentation, was not reviewed at this Inspection.

SCD, on August 21, 1991, submitted a Part A permit application and pre-compliance certification under the Boiler and Industrial Furnace (BIF) regulations for boiler No. 3. SCD had a discharge from one of its collection sumps that allowed chlorinated benzenes to be released to the environment and contaminated the ground water. Under the DNREC Consent Order No. 88-11 and appropriate permits SCD operates an air stripper to remove the contamination from the ground water. The vapor stream from this air stripper is vented to the No. 3 boiler for thermal treatment and destruction. Though SCD does not feel that this waste stream meets the definition of hazardous waste they submitted this application to EPA to preserve their Interim Status under BIF should EPA rule that this waste stream is a solid and hazardous waste.